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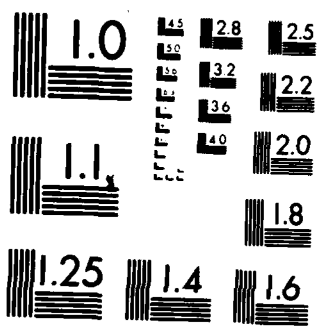

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THE POLARIZATION RATIO OF CRYSTAL MONOCHROMATORS

LAURENCE D. JENNINGS
MATERIALS CHARACTERIZATION DIVISION

June 1983

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ABSTRACT

A tabulation of some 40 measured values of the polarization ratio K (a measure of the fractional polarization introduced into an X-ray beam by a crystal monochromator) is presented. The values may be represented through the parameter n given by $K = \cos^n 2\theta_M$. The measured values of n cluster around unity and the theoretical rationale for this result is discussed. Possible explanations of outlying values are considered. It is recommended that the polarization ratio of an apparatus be measured using a direct method whenever possible, but methods for estimating a value of K are also given.

INTRODUCTION

X-ray diffraction is used in applications ranging from determination of the structure of magnetic materials to determination of residual stress in formed metal parts. When such measurements are required at the highest levels of sensitivity, the polarization ratio is a required parameter. Thus a study of the polarization ratio will lead to a better usage of X-ray diffraction methods such as those mentioned.

Typically the beam in a crystal monochromated X-ray diffraction experiment is partially polarized, and the degree of polarization must be known in order to use the correct polarization factor in the interpretation of the data. Many authors tacitly assume that the degree of polarization may be adequately estimated by considering that the crystal monochromator acts as an ideally mosaic diffractor placed in an otherwise unpolarized beam of characteristic radiation. Actually, most measurements have shown that this assumption is not correct and that the deviation from its prediction is significant by modern standards of accuracy in the case of radiation of wavelength greater than about 1 Å. It is clearly of some importance to establish whether these measurements are not typical of diffraction apparatuses or whether those crystallographers making use of the tacit assumption should reassess their procedures. Accordingly, the Commission on Crystallographic Apparatus of the International Union of Crystallography (IUCr, 1978) instituted a survey of polarization ratios. The call for response to the survey offered additional material on measuring, understanding, and reporting polarization ratios. At about the same time, LePage, Gabe and Calvert[1] published a simple technique for measuring polarization ratios, making this information available in a widely read crystallographic journal. In spite of this activity, there was very little response to the survey, which was extended through the IUCr 1981 Congress, at which additional invitations to respond were proffered. These did bring forth additional responses, and it is therefore now appropriate to publish all the information which has come to the attention of the survey organizer. We will give a short discussion of polarization ratios, sufficient to understand the reported values, then a table of results, and finally some brief comments on the values.

DISCUSSION OF POLARIZATION RATIOS

We may distinguish a beam polarization ratio K from a sample polarization ratio α. This latter may be used to characterize specimen perfection as exemplified by the work of Chandrasekhar and coworkers[2] and by the extensive research of Olekhnovich and associates,[3] unfortunately mostly on semi-conducting materials rather than on typical monochromating material. It is convenient to distinguish two meaningful cases of sample polarization ratios. At one extreme, we have the ratio of the reflectivity of the sample for a well-collimated beam of each of the two polarizations. This parameter may be called α_δ (because the angular distribution of the beam is a δ-function), and is clearly a function of the angular setting of the sample. The quantity α_δ may be called a polarization coefficient or the reflectivity polarization ratio. If the angular setting of the sample (of monochromating material) is not specified, it may be assumed that the polarization coefficient at maximum reflecting power is being quoted. At the other extreme, we may consider the integrated polarization ratio given by $\alpha_\rho = \rho_\parallel/\rho_\perp$, the ratio of the two integrated intensities.

1. LePAGE, Y., GABE, E. J., and CALVERT, L. D. *X-Ray Beam Polarization Measurements.* J. Appl. Cryst., v. 12, 1979, p. 25-26.
2. CHANDRASEKHAR, S., RAMASESHAN, S., and SINGH, A. K. *Experimental Determination of the Extinction Factor by the Use of Polarized X-Rays.* Acta Cryst., v. A25, 1969, p. 140-142.
3. OLEKHNOVICH, N. M., KARPEI, A. L., and MARKOVICH, V. L. *Polarization of Mo K_α-Radiation of the Bragg Diffraction in Real Silicon Crystals.* Krist. Tech., v. 13, no. 12, 1978, p. 1463-1469.

The beam polarization ratio is not a property of a material, but rather of an apparatus. For an arrangement with no polarization dependent components after the sample, it is the ratio of the effective power (incident on the sample) in each of the two polarization states. In principle, because of non-uniformity in the beam and angularly dependent absorption effects in the sample, the beam polarization ratio might not be independent of diffractometer settings. In practice, a constant value is usually assumed. In the case that the experimental beam is prepared by diffracting a well-collimated, unpolarized source beam from a monochromator with a polarization coefficient α_δ, we would find that the experimental beam has a polarization ratio $K_\delta = \alpha_\delta$. If, on the other hand, the source beam had a uniformly illuminated broad angular distribution, we would have $K_\rho = \alpha_\rho$. The optimum geometry for maximum monochromated power usually is intermediate between these extremes. Thus the observed value of K, even for an unpolarized source beam, is given by some sort of an averaged sample polarization ratio α. Because the appropriate weighting function has no particular significance except with respect to an individual apparatus, this value of α does not effectively characterize the monochromator material. Usually, however, α would be expected to lie between α_δ and α_ρ and thus knowledge of these two extremes would delimit the possible range of α and hence of K (for an unpolarized source beam).

Similar reasoning applies in the case of a diffracted beam monochromator. The relevant value of α would involve a weighting function that depends on the angular and spacial distribution of the beam incident on the monochromator. These distributions are more likely to depend on diffractometer settings than they are in the case of an incident beam monochromator. Nevertheless, to some approximation, we may define an effective apparatus polarization ratio, conventionally also called K. For an unpolarized incident beam, K would still be expected to lie between α_δ and α_ρ.

Values of K can be measured by a number of different methods, which can be broadly characterized as "direct" or "indirect." The direct methods are those which give the beam polarization ratio directly as the quotient of two measured quantities. Several of these methods are discussed by Suortti and Jennings,[4] but the most convenient one for 0.5 - 1% accuracy is that described by LePage et al[1] and in the appendices to the IUCr announcement[5] (still available from L. D. Jennings). This method makes use of an amorphous sample scattering at 90° in each of two orthogonal planes. Direct methods are more cumbersome for the apparatus polarization ratio in the case of a diffracted beam monochromator; although each of the two polarizations can be selected with a Borrmann polarizer or 90° scattering, it is difficult to make the divergence conditions identical in each of the two measurements.

Indirect methods require more complicated analysis. For example, one can infer approximate values of an apparatus polarization ratio from measurements of the integrated intensity of the monochromator or of the two extremes of its sample polarization ratio. However, the only indirect method used in any of the work reported here is the comparison method introduced by Miyake, Togawa and Hosoya.[6] Their technique is to compare relative integrated intensities obtained with filtered, presumably unpolarized, characteristic radiation to those obtained with a monochromated apparatus with unknown polarization ratio. This method requires accurate

4. SUORTTI, P., and JENNINGS, L. D. *International Union of Crystallographic Apparatus Accuracy of Structure Factors from X-Ray Powder Intensity Measurements.* Acta Cryst., v. A33, Part 6, 1977, p. 1012-1027.
5. IUCr. *Polarization Ratio for X-Rays - A Survey by the Commission on Crystallographic Apparatus.* Acta Cryst., v. A34, Part 1, 1978, p. 159-160.
6. MIYAKE, S., TOGAWA, S., and HOSOYA, S. *Polarization Factor for X-Ray Monochromator Crystals.* Acta Cryst., v. 17, 1964, p. 1083-1084.

comparison of integrated intensities using different background subtraction techniques and also knowledge of the extinction properties of the sample. The K value is obtained by letting it be a parameter determined by a least squares fit to the comparison. Although Vincent and Flack[7] have recently supported the use of this technique, the difficulties in its implementation have been emphasized by Mathieson[8] and by DeMarco et al.[9]

THE APPARATUS COMMISSION SURVEY

The IUCr survey was specifically directed toward K values. Therefore, all measured K values known to the author are entered in Table 1. In addition, the above discussion shows that α values are of substantial interest in assessing the expected range of K values. Unfortunately, most measurements of α have been on materials which are not customarily used as monochromators. A few values for the important practical case of graphite at CuKα are given in Table 1. Insofar as the information is available, the table indicates whether the monochromator was before or after the sample and whether a direct or comparison method was employed. Further useful information was available in so few cases that it did not seem worthwhile to include it.

When polarization ratios at various wavelengths are considered, it is convenient to define a parameter n through the relation $K = \cos^n 2\theta_M$, where θ_M is the monochromator Bragg angle. We may similarly characterize a sample polarization ratio through $\alpha = \cos^m 2\theta$. The n or m values are listed in Table 1. Clearly the constraints on values of polarization ratio could equally well be discussed in terms of the n and m values.

Various available extinction theories yield a relationship between m and the extinction coefficient y, as shown, for example, in the papers of Jennings.[10,11] All theories limit the range of m from zero to two; this result clearly applies to the n values of apparatus polarization ratios if the only polarizing component is a crystal monochromator. Furthermore, if the y value for the monochromator were known, the various theories suggest a comparatively limited possible range of m values. Unfortunately, very few integrated intensities for monochromators have been reported, but the work of Jennings[10,11] and of Lawrence[12] suggests a typical range of y from 0.3 to 0.4. For graphite in symmetrical reflection at CuKα, the theories considered by Jennings (Ref. 11, Figs. 2 and 3) give corresponding m values from 0.8 to 1.2. This result is not much changed for other typical monochromators at crystallographic wavelengths (Jennings, Ref. 10, and unpublished results).

The n and m values of Table 1, for the most part, lie near this expected range, 0.8 to 1.2, supporting the theoretical reasoning. We will therefore make some general remarks using the language of these theories and then consider specifically some of the entries in Table 1 which illustrate significant points.

7. VINCENT, M. G., and FLACK, H. D. *On the Polarization Factor for Crystal-Monochromated X-Radiation. II. A Method for Determining the Polarization Ratio for Crystal Monochromators.* Acta Cryst., v. A36, Part 4, 1980, p. 614-620.
8. MATHIESON, A. McL. *A Comment on the Method of Determination of the Polarization Ratio for Crystal-Monochromated X-Rays by Vincent & Flack.* Acta Cryst., v. A38, Part 5, 1982, p. 739-740.
9. DeMARCO, J. J., JENNINGS, L. D., MAZZONE, G., and SACCHETTI, F. *Assessment of Experimental Methods for Measuring X-Ray Polarization Ratios.* Comitato Nazionale Energia Nuleare (Rome) Report CNEN-RT/FI(81)22, 1981.
10. JENNINGS, L. D. *Polarization of Crystal Monochromated X-Rays.* Acta Cryst., v. A24, 1968, p. 472-474.
11. JENNINGS, L. D. *Extinction, Polarization and Crystal Monochromator.* Acta Cryst., v. A37, Part 4, 1981, p. 584-593.
12. LAWRENCE, J. L. *The Reflectivity of a Pyrolytic Graphite Monochromator.* Acta Cryst., v. A38, Part 6, 1982, p. 859-863.

Table 1. MEASURED VALUES OF POLARIZATION RATIOS

A = monochromator after the sample (in the diffracted beam). B = monochromator before the sample (in the incident beam). C = comparison method. D = a direct method. K is an apparatus polarization ratio, characterized by the parameter n, and α is a sample polarization ratio, characterized by m, as detailed in the text.

Entry Number	Conditions	K or	n or m	Reference
		Graphite @ CrKα; cos 2θ = 0.766		
1	A,C	0.75(4)	1.08	Altree-Williams and Jordan, Ref. 14
		Graphite @ CoKα; cos 2θ = 0.857		
2	B,D	0.919(4)	0.54	DeMarco et al, Ref. 9
		Graphite @ CuKα; cos 2θ = 0.894		
3	B,C	0.860(14)	1.35	Vincent and Flack, Ref. 7
4	C	0.86	1.35	Hope, Ref. 15
5	D	0.89	1.04	Sparks, Ref. 16
6	B,D	0.896(6)	0.98	Annaka, S., Personal Communication, 1981
7	B,D	0.897(5)	0.97	LePage, Gabe, and Calvert, Ref. 1
8	B,D	0.905	0.89	Suortti and Jennings, Ref. 4
9	A,D	0.906(8)	0.88	Valvoda, V., Personal Communication, to L. D. Calvert, 1981
10	B,D	0.908(5)	0.86	LePage, Gabe, and Calvert, Ref. 1
11	B,D	0.925	0.70	Suortti and Jennings, Ref. 4
12	A,C	0.93(4)	0.65	Altree-Williams and Jordan, Ref. 14
13a	B,D	0.985	0.13	Cohen, J. B., Personal Communication, 1982
13b	B,D	0.989	0.10	Bardhan and Cohen, Ref. 17
14a	α_δ	0.905(14)	0.89	Calvert, Killean, and Mathieson, Ref. 18
14b	α_δ	0.888(18)	0.99	Calvert, Killean, and Mathieson, Ref. 19
15	α_ρ	0.803	1.96	Olekhnovich et al, Ref. 13
16	α_ρ	0.899	0.95	Ref. 13
		Graphite @ MoKα; cos 2θ = 0.978		
17	B,C	0.907(11)	4.29	Vincent and Flack, Ref. 7
18	B,D	0.970(3)	1.34	LePage, Gabe, and Calvert, Ref. 1
19a	B,D	0.969(3)	1.42	Ref. 1
19b	B,D	0.973(5)	1.20	Ref. 1
		Graphite @ AgKα; cos 2θ = 0.986		
20	B,C	0.805(11)	15.4	Vincent and Flack, Ref. 7
21	B,D	1.000(2)	0.0	DeMarco et al, Ref. 9
		LiF @ CoKα; cos 2θ = 0.605		
22	B,D	0.60(2)	1.02	Suortti, P., Personal Communication, 1983

14. ALTREE-WILLIAMS, S., and JORDAN, B. *Polarization Ratio of a Diffracted-Beam Monochromator in X-Ray Powder Diffractometry.* Anal. Chem., v. 52, no. 8, 1980, p. 1296-1300.
15. HOPE, H. *Polarization Factor for Graphite X-Ray Monochromators.* Acta Cryst., v. A27, 1971, p. 392-393.
16. SPARKS, C. J. *Excess Diffuse X-Ray Scattering and Anomalous Dispersion* in Anomalous Scattering, ed. S, Ramaseshan and S. C. Abrahams, 1974, p. 175-192; also in Proceedings of IUCr Conference, Madrid.
17. BARDHAN, P., and COHEN, J. B. *A Structural Study of the Alloy Cu_3Au Above its Critical Temperature.* Acta Cryst., v. A32, 1976, p. 597-614.
18. CALVERT, L. D. KILLEAN, R. C. G., and MATHIESON, A. McL. *Polarization Ratios of a Pyrolytic Graphite Crystal for $CuK_{\alpha 1}$ X-Rays* in International Crystallography Conference on Diffraction Studies of Real Atoms and Real Crystals (Extended Abstracts), Australian Academy of Sci., Canberra, Australia, 1974, p. 88-89.
19. CLAVERT, L. D., KILLEAN, R. C. G., and MATHIESON, A. McL. *The Measurement of the Polarization Ratio for X-Rays and the Use of Polarized X-Rays.* Annual Report 1973-74 of Division of Chemical Physics, CSIRO, Clayton, Australia, 1974, p. 24-26.

Table 1. CONTINUED

		LiF @ CuKα; cos 2θ = 0.707		
23	B,D	0.62(1)	1.38	Reid, J. S., Personal Communication, 1981
24	B,C	0.624	1.36	Miyake, Togawa, and Hosoya, Ref. 6
25	B,D	0.629	1.34	Colella and Batterman, Ref. 20
26	D	0.65	1.24	Sparks, Ref. 16
27	B,D	0.664(5)	1.18	Trucano, P., and Batterman, B. W.*
28	D	0.69	1.07	Sparks, Ref. 16
29	B,D	0.707(7)	1.00	Walker, C. B., Personal Communication, 1977
30	B,D	0.722(2)	0.94	Jennings, Ref. 10
31	B,D	0.730(6)	0.91	Annaka, S., Personal Communication, 1981
32	B,D	0.730	0.91	Suortti and Jennings, Ref. 4
33	B,D	0.780	0.72	Ref. 4
		LiF @ MoKα; cos 2θ = 0.938		
34	B,D	0.93(1)	1.13	Suortti, P., Personal Communication, 1983
35	D	0.944(2)	0.90	Reid, J. S., Personal Communication, 1981
36	B,D	0.96	0.63	Chipman, D P., and Jennings, L. D., Unpublished Measurements
		Quartz @ CuKα; cos 2θ = 0.894		
37	C	0.825	1.71	Hosoya, S.*
38	A,C	0.90(2)	0.94	Linkoaho, Rantavuori, and Korhonen, Ref. 21
39	B,D	0.905	0.89	Suortti and Jennings, Ref. 4
40	B,D	0.915	0.79	Ref. 4
41		0.94(2)	0.55	Stephan and Löschau, Ref. 22
42		0.95(2)	0.46	Ref. 22
		Germanium @ CuKα; cos 2θ = 0.888		
43	D	0.94(2)	0.52	Olekhnovich, Ref. 23

Entries 8 and 11 represent measurements on the same material; the former is more nearly K_ρ, the latter more nearly K_δ.

Entry 13a is an alternate, recent measurement of the apparatus of entry 13b, as discussed in the text.

Entry 14a was not corrected for secondary extinction and is thus comparable to the other entries; 14b is the same data corrected for secondary extinction.

Entries 15 and 16 represent the extreme cases of the 12 samples studied.

Entry 19a includes both characteristic and continuum radiation, and is thus comparable to the other entries; 19b is the same data with the continuum removed.

The following pairs of entries represent different specimens studied in otherwise nearly identical conditions: 7 and 10; 15 and 16; 18 and 19; 26 and 28; 32 and 33; and 39 and 40.

*Response to IUCr Powder Intensity Project, 1968.

20. COLELLA, R., and BATTERMAN, A. W. *X-Ray Determination of Phonon Dispersion in Vanadium.* Phys. Rev., v. B1, no. 10, 1970, p. 3913-3921.
21. LINKOAHO, M, RANTAVUORI, E., and KORHONEN, U. *Supplement to the Powder Intensity Project of the IUCr.* Acta Cryst., v. A27, 1971, p. 495-496.
22. STEPHAN, D., and LÖSCHAU, W. *Zum Reflexionsvermögen und Polarisationsverhältnis bei der Röntgenstrahlbeugung am Realkristall (I) Experimentelle Ergebnisse und Vergleich mit der Theorie.* Krist. Tech., v. 11, no. 12, 1976, p. 1295-1301.
23. OLEKHNOVICH, N. M. *The Polarization Factor in X-Ray Scattering, Taking Into Account Monochromator Extinction.* Soviet Physics - Crystallography, v. 14, no. 2, 1969, p. 203-206; also in Kristallografiya, v. 14, p. 261-265.

In many geometries, it is desirable to arrange the monochromator for maximum reflecting power. In general such an arrangement minimizes the extinction coefficient y and leads to n values near to or less than unity. (This does not imply that the monochromator is a nearly perfect crystal; this result holds true because of sizable secondary extinction.) Some monochromators may not be adjusted for high reflectivity, and in these cases n values near 2 are perfectly plausible. In any case, a few workers studied monochromator materials with varying rocking widths; the trend toward smaller n values with narrowing rocking curves (higher reflecting power, smaller extinction coefficient) is unmistakable, though there is great variation from sample to sample (Suortti and Jennings;[4] LePage et al;[1] and, especially, Olekhnovich et al[13]).

The only other trend observed is that the comparison method gave, on the average, higher n values than the direct methods. No information is available on whether the apparatuses studied with the comparison method, on the average, used less efficient monochromators, or whether there is a shortcoming in one of the methods.

It is of interest to examine, in Table 1, each of the entries which is outside the plausible range of n values. Entry 21 illustrates the important point that a small amount of continuum is generally included as part of the "monochromatic" beam. Suppose that the sample polarization ratio of the monochromator is given by $\cos^m 2\theta_M$, that the fraction of the beam power arising from the continuum is f, and that the fractional polarization of the continuum is P. It is easy to show that

$$K = \cos^m 2\theta \; \frac{1 - fP}{1 + fP}.$$

The apparent low n value for the beam polarization ratio of Entry 21 may then be explained by a plausible choice of the parameters, such as m = 0.5, P = -0.25, and f = 0.014 or m = 1.0, P = -0.25, and f = 0.028. It is somewhat of a problem to measure any of these parameters independently. However, the suggested values of m are reasonable. As has been discussed already, the value of P is estimated from published data and its sign from the knowledge that the exciting electron beam is in the plane of diffraction, and the values of f are consistent with dispersive scans of the wavelength distribution of the beam. From this discussion, it can be seen that the measurement of the beam polarization ratio does not accurately determine the m value characterizing the sample polarization ratio in this case. This situation comes about because of the small possible range of polarization ratios and from the presumably greater continuum contamination for hard radiation. For softer radiations, the n values of Table 1 probably characterize the sample polarization ratios reasonably well. Furthermore, by using special techniques, the continuum can be subtracted as part of the background (Entry 19b), but such procedures are not usually employed.

The original measurement, Entry 13b, was carried out using a different radiation from that used in the actual experiment. The later measurement, Entry 13a, giving substantially the same result, was carefully carried out without disturbing the experimental arrangement. The apparatus uses quite large divergences, and it may be that a relatively large amount of continuum was included, making the above discussion of small n values applicable.

13. OLEKHNOVICH, N. M., MARK[illegible]CH, V. L., [illegible]KHNOVICH, A. N., and POLUCHANKINA, L. P. *The Measurement of the Polarization Characteristics of an [illegible] Diffr[illegible]n Beam.* Izvestiya Akademii Nauk BSSR, no. 2, 1981, p. 64-67.

The measurements of Entries 17 and 20 are stated to be of high accuracy and were the primary objective of the experimental program. Unfortunately the authors do not state the geometrical parameters, do not report having verified the unusual results with a direct method, and do not give a rationale for the results. It may be relevant that the experiments were carried out with a sample displaying higher extinction than in other implementations of the comparison method. It remains to be established with certainty whether there is some considerable difficulty in the application of the comparison method, as suggested by Mathieson,[8] or whether some physical principles, unclear to this author, must be considered.

CONCLUSIONS AND RECOMMENDATIONS

In summary, the author offers these recommendations based on the results shown in Table 1 and on other experience.

1. For accurate work, especially for radiations of wavelength greater than 1 Å, the polarization ratio of a typical crystal monochromated apparatus must be established.

2. If at all possible, measure the apparatus polarization ratio K using a direct method.

3. If such a measurement is not feasible, a reasonable first guess is that $K = \cos^n 2\theta_M$, with $n = 1$. This is not tantamount to an assumption that the monochromating crystal is nearly ideally perfect.

4. If the set-up is of relatively high efficiency, lower the n value somewhat; this situation is valid for a relatively narrow crystal rocking curve and/or good collimation conditions (as typically apply to a bent monochromator). Conversely, raise the n value somewhat for a low efficiency set-up.

5. Establish from the geometry whether the continuum contribution increases or decreases K. (The polarization is along the exciting electron beam.) The meager information available suggests that a typical magnitude for this change in K is about 0.01 (assuming, of course, that the predominant component of the beam is initially unpolarized characteristic radiation).

6. Procedures for dealing with polarization ratios are not well established. Therefore a publication listing a polarization ratio should detail relevant geometrical aspects and the methods used for determining the ratio, as well as its value.

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